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583 ANSWERS

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=> d l18 1-5 ibib abs hitstr hitind

L18 ANSWER 1 OF 5 HCAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER:

2003:696276 HCAPLUS

TITLE:

Process for the preparation of hydrogen

peroxide

INVENTOR(S):

Borthakur, Naleen

PATENT ASSIGNEE(S):

India

SOURCE:

U.S. Pat. Appl. Publ., 7 pp.

CODEN: USXXCO

DOCUMENT TYPE:

Patent English

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.

KINDDATE APPLICATION NO. DATE

US 2003165421 A1 20030904 US 2001-24205 20011221 PRIORITY APPLN. INFO.: US 2001-24205 20011221

The invention provides a process for the prepn. of hydrogen peroxide by hydrogenating 3-2-(oxopropyl)-2(1H)-quinoxalinone in the presence of a palladium catalyst and contacting the 3-2-(oxopropyl)-1,2,3,4-tetrahydro-2-quinoxalinone with oxidant mol. oxygen or air in ethylacetate-water or chloroform-water biphasic system.

1333-74-0, Hydrogen 24949-44-8 39260-15-6 106511-13-1 106511-14-2 127443-93-0 273196-02-4 592534-84-4 592534-85-5

(process for prepn. of hydrogen peroxide)

RN 1333-74-0 HCAPLUS

CN Hydrogen (8CI, 9CI) (CA INDEX NAME)

н— н

RN 24949-44-8 HCAPLUS CN 2(1H)-Quinoxalinone, 3,4-dihydro-3-(2-oxopropylidene)- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} H & O \\ \parallel & \\ N & CH-C-Me \end{array}$$

RN 39260-15-6 HCAPLUS CN 2(1H)-Quinoxalinone, 3,4-dihydro-3-(2-oxo-2-phenylethylidene)- (9CI) (CA INDEX NAME)

RN 106511-13-1 HCAPLUS CN 2(1H)-Quinoxalinone, 3,4-dihydro-1-methyl-3-(2-oxopropylidene)-(9CI) (CA INDEX NAME)

RN 106511-14-2 HCAPLUS

CN 2(1H)-Quinoxalinone, 3,4-dihydro-3-(2-oxopropyl)- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} H & O \\ \parallel & \parallel \\ N & CH_2-C-Me \end{array}$$

RN 127443-93-0 HCAPLUS

CN 2(1H)-Quinoxalinone, 3,4-dihydro-3-(2-oxobutylidene)- (9CI) (CA INDEX NAME)

RN 273196-02-4 HCAPLUS

CN 2(1H)-Quinoxalinone, 3,4-dihydro-3-(2-oxo-2-phenylethyl)- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} H & O \\ N & CH_2 - C - Ph \\ N & O \end{array}$$

RN 592534-84-4 HCAPLUS

CN 2(1H)-Quinoxalinone, 3,4-dihydro-3-(2-oxobutyl)- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} & O & \\ & \parallel & \\ N & CH_2-C-Et \\ & N & O \end{array}$$

RN 592534-85-5 HCAPLUS CN 2(1H)-Quinoxalinone, 3,4-dihydro-1-methyl-3-(2-oxopropyl)- (9CI) (CA INDEX NAME)

IT 7722-84-1P, Hydrogen peroxide

(process for prepn. of hydrogen peroxide)

RN 7722-84-1 HCAPLUS

CN Hydrogen peroxide (H2O2) (9CI) (CA INDEX NAME)

HO-OH

IC ICM C01B015-022

NCL 423587000

CC 49-8 (Industrial Inorganic Chemicals)

ST prepn hydrogen peroxide

IT Hydrogenation

Hydrogenation catalysts

Solvents

(process for prepn. of hydrogen peroxide)

IT 7440-44-0, Carbon

(activated; process for prepn. of hydrogen

peroxide)

IT 7440-05-3, Palladium

(process for prepn. of hydrogen peroxide)

IT 1333-74-0, Hydrogen 7664-93-9, Sulfuric acid 7782-44-7, Oxygen 24949-44-8 39260-15-6 106511-13-1

106511-14-2 127443-93-0 273196-02-4 592534-84-4 592534-85-5

(process for prepn. of hydrogen peroxide)

7722-84-1P, Hydrogen peroxide IT

(process for prepn. of hydrogen peroxide)

75-09-2, Dichloromethane 67-66-3, Chloroform 71-43-2, Benzene TI141-78-6, Ethyl acetate 75-65-0, Tert-Butyl alcohol (process for prepn. of hydrogen peroxide)

HCAPLUS COPYRIGHT 2003 ACS on STN ANSWER 2 OF 5 L18

ACCESSION NUMBER:

1993:580281 HCAPLUS

DOCUMENT NUMBER:

119:180281

TITLE:

Spectral characteristics of the reaction products of 5-phenyl-2,3,4-furantrione with

o-diamines

AUTHOR(S):

Rashed, Nagwa; Mousaad, Ahmed; Moussa, Adel; El

Ashry, El Sayed H.

CORPORATE SOURCE:

Fac. Sci., Alexandria Univ., Alexandria, Egypt Spectroscopy Letters (1993), 26(6), 975-95

SOURCE:

CODEN: SPLEBX; ISSN: 0038-7010

DOCUMENT TYPE:

Journal English

LANGUAGE:

GΙ

- * STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY AVAILABLE VIA OFFLINE PRINT *
- The 1H and 13C NMR and mass spectra of 2-(2-amino-4,5-AΒ dimethylphenylcarbamoyl)-3-(hydroxyphenylmethyl)-6,7dimethylquinoxaline, 3-(hydroxyphenylmethyl)-6,7-dimethylquinoxalin-2-carboxylic-.gamma.-lactone, 3-(hydroxyphenylmethyl)-6,7dimethylquinoxalin-2-carboxylic acid phenylhydrazide, 3-[2-hydroxy-2-phenyl-1-(phenylhydrazono)ethyl]-6,7-dimethyl-2(1H)quinoxalinone, 2,3-dihydro-6,7-dimethyl-3-phenylhydrazono-2phenylfuro[2,3-b]quinoxaline, 3-(hydroxyphenylmethyl)-6,7-dimethyl-1phenylflavazole, and 3-(acetoxyphenylmethyl)-6,7-dimethyl-1phenylflavazole (I-VII, resp., R = Me) have been studied.

IT 1333-74-0

(nuclear magnetic resonance, of quinoxaline, quinoxalinone, furoquinoxalinone, and flavazole derivs., proton and carbon-13)

1333-74-0 HCAPLUS RN

Hydrogen (8CI, 9CI) (CA INDEX NAME) CN

H-H

150240-27-0P TT

(prepn. and spectra of)

RN150240-27-0 HCAPLUS

2(1H)-Quinoxalinone, 3-[2-hydroxy-2-phenyl-1-(phenylhydrazono)ethyl]-CN6,7-dimethyl-, (E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.

22-10 (Physical Organic Chemistry) CC Section cross-reference(s): 33

14762-74-4 1333-74-0 IT

(nuclear magnetic resonance, of quinoxaline, quinoxalinone, furoquinoxalinone, and flavazole derivs., proton and carbon-13)

150240-26-9P **150240-27-0P** 150240-25-8P 150240-24-7P IT150240-30-5P 150240-29-2P 150240-28-1P

(prepn. and spectra of)

HCAPLUS COPYRIGHT 2003 ACS on STN ANSWER 3 OF 5 L18

ACCESSION NUMBER:

1988:112397 HCAPLUS

DOCUMENT NUMBER:

108:112397

TITLE:

Heterocycles from carbohydrate precursors.

A novel synthesis of pyridazinones.

Preparation of 3-[1-aryl-6(1H)-pyridazinon-3-yl]-

2(1H)-quinoxalinones

AUTHOR(S):

El Ashry, El Sayed H.; El Kilany, Yeldez; Amer,

Adel

CORPORATE SOURCE:

SOURCE:

Fac. Sci., Alexandria Univ., Alexandria, Egypt

Heterocycles (1987), 26(8), 2101-8

CODEN: HTCYAM; ISSN: 0385-5414

DOCUMENT TYPE:

LANGUAGE:

OTHER SOURCE(S):

Journal English

CASREACT 108:112397

GΙ

Wittig olefination of quinoxalinone derivs. I (R = Ph, 4-MeC6H4, 2-MeC6H4, R1 = CHO) with Ph3P:CHCO2Et gave adducts I [R1 = (E)-CH:CHCO2Et] (II) in 70-76% yields. Thermal isomerization of II gave pyrazonylquinoxalinones III, as did Wittig olefination of I (R = 4-ClC6H4, R1 = CHO) or olefination of I (R = Ph, R1 = CHO) at 155.degree.. Sapon. of II (R = Ph) gave a 1:3 mixt. of III (R = Ph) and pyrazologuinoxaline IV.

IT 113314-02-6

(oxidative cleavage of, with sodium periodate)

RN 113314-02-6 HCAPLUS

CN 2(1H)-Quinoxalinone, 3-[2,3,4-trihydroxy-1-[(2-methylphenyl)hydrazono]butyl]-, [S-(R*,R*)]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry unknown.

CC 28-18 (Heterocyclic Compounds (More Than One Hetero Atom))

IT 113314-02-6

(oxidative cleavage of, with sodium periodate)

IT 113314-13-9P

(prepn. and catalytic hydrogenation of)

HCAPLUS COPYRIGHT 2003 ACS on STN ANSWER 4 OF 5

ACCESSION NUMBER: 1961:143616 HCAPLUS

55:143616 DOCUMENT NUMBER:

AUTHOR(S):

SOURCE:

AΒ

CORPORATE SOURCE:

55:27082i,27083a-i,27084a-i,27085a-i,27086a-h ORIGINAL REFERENCE NO.: Amino sugar syntheses. XIX. Base-catalyzed TITLE:

transformations of N-phenyl-D-hexosaminic [2-deoxy-2-phenylamino-D-hexonic] nitriles

Kuhn, Richard; Weiser, Dieter; Fischer, Hans

Max-Planck Inst., Heidelberg, Germany

Ann. (1959), 628, 207-39

Journal

DOCUMENT TYPE: LANGUAGE: Unavailable

For diagram(s), see printed CA Issue. GΙ

Reaction of a D-pentose with an arylamine (RNH2) and HCN gave two epimers of 2-arylamino-2-deoxy-D-hexonic nitrile (I); all 8 of these were prepd. Each I was transformed stepwise, by the action of dil. alkali, to the resp. 2-arylamino-1,2-dideoxy-1-imino-D-hexofuranose (II), 2-arylamino-2-deoxy-D-hexo-1-ene-furanosylamine (III), and thence, by loss of H2O, to 2-arylamino-1,2,3-trideoxy-1-imino-D-hexo-2-ene-furanose (IV). The resp. IV was used for the prepn. of the 2-amino-2,3-dideoxy-D-hexose (V) and the 3-deoxy-D-hexulosonic acid (VI). [For various I, II, III, and IV, configuration and substituent are indicated]. I (D-galacto, R = Ph) (VII) (3 g.) in 20 cc. MeOH was treated at 20.degree. with 0.1 g. KOH in 2 cc. MeOH, the mixt. kept 10 min., and the crystals removed and washed with EtOH to give 2.3 g. II (D-galacto, R = Ph) (VIII), m. 121.degree. (decompn.), [.alpha.]20D -49.8.degree. (2 min.; c 0.8, MeOH). For prepg. VIII ag. NaOH or ag. Ba(OH)2 may also be used. I (D-talo, R = Ph) (IX) (2 q.) in 8 cc. MeOH was similarly treated 20 min. with 0.3 cc. 10% KOH in MeOH to give 1.5 g. VIII, m. 121.degree.. (D-ido, R = Ph) (X) (6 g.) was suspended in 20 cc. MeOH at 20.degree., treated with 400 mg. KOH in 10 cc. MeOH, the mixt. shaken 15 min. (clear soln.), kept 5 min., and the crystals removed, to give 4 g. III (D-xylo, R = Ph) (XI), m. 122.degree. (decompn.), [.alpha.]20D -3.6.degree. (3 min.; c 0.42, MeOH). The same product resulted by action of NH3, PhCH2NH2, or Et3N in MeOH, or of aq. Similar treatment of I (D-gulo, R = Ph) (XII) gave a mixt. (XIII), m. 127-33.degree., of XI with IV (D-threo, R = Ph) (XIV); recrystn. of XIII gave pure XIV. X (10 g.) in 500 cc. MeOH was treated 15 min. with 0.6 g. KOH in 20 cc. MeOH, the suspension of XI heated 20 min. at 50.degree., and the soln. cooled and evapd. to 100 cc. to give 5.2 g. XIV, m. 147.degree. (MeOH-petr. ether), [.alpha.]20D -122.degree. (c 0.54, MeOH), reducing to Fehling soln., no NH2 (Van Slyke). Treatment of X with PhNH2 in MeOH (2 days) gave A soln. of 15 g. VIII in 200 cc. MeOH was heated 30 min. at 50-55.degree., kept 20 hrs. at room temp., evapd. in vacuo to 50 cc., and cooled to -5.degree. to give 9.3 g. XIV, m. 146-7.degree.. IIIA (2.3 g.) in 15 cc. MeOH was kept 20 min. at 50-60.degree. and 15 hrs. at 20.degree., and treated with petr. ether to give 1.4 g. XIV. I (Dgulo, R = p-MeC6H4) (XV) in MeOH (c 1.16), on addn. of 0.35 mg. KOH/cc., showed [.alpha.]18D 160.degree. (2 min.) .fwdarw. 111.degree. (10 min.) .fwdarw. 39.degree. (25 min.) .fwdarw.

-7.1.degree. (40 min.) .fwdarw. -50.degree. (60 min.) .fwdarw. -104.degree. (5 hrs.) .fwdarw. -107.degree. (7 hrs., const.). IX in MeOH (c 1.03), on addn. of 0.33 mg. KOH/cc., showed [.alpha.]20D -135.degree. (4 min.) .fwdarw. -80.degree. (20 min.) .fwdarw. -30.degree. (41 min.) .fwdarw. -14.degree. (60 min.) .fwdarw. -12.degree. (87 min.) .fwdarw. -24.degree. (160 min.) .fwdarw. -56.degree. (6 hrs.) .fwdarw. -106.degree. (24 hrs., const.). I (D-gluco, R = Ph) (XVI) (15 g.) in 100 cc. MeOH was treated with 0.5 q. KOH, the soln. kept 20 hrs. at room temp. and evapd. in vacuo, the sirup stirred with 150 cc. H2O, and the crystals recrystd. (MeOH-H2O) to give 10 g. IV (D-erythro, R = Ph) (XVII), m. 109-10.degree., [.alpha.]20D 74.8.degree. (c 1.38, MeOH), reducing to Fehling soln. I (D-attro, R = Ph) (XVIII) (1 g.), similarly treated, gave 0.6 g. XVII, m. 110.degree. I (D-manno, R = Ph) (XIX) was isolated in small yield from the mother liquor from the prepn. of XVI (from D-arabinose, PhNH2, and HCN) by repeated pptn. with EtOH and petr. ether, [.alpha.]20D -102.degree. (c 0.61, MeOH). in MeOH (c 0.61), on addn. of 0.67 mg. KOH/cc., showed [.alpha.]20D -90.degree. (2 min.) .fwdarw. -55.degree. (5 min.) .fwdarw. -6.5.degree. (10 min.) .fwdarw. 16.4.degree. (14 min.) .fwdarw. 56.degree. (24 min.) .fwdarw. 67.degree. (1 hr., const.). I (D-allo, R = Ph) (XX) in MeOH, [.alpha.]20D 148.degree. (c 0.44), on addn. of 0.33 mg. KOH/cc., showed [.alpha.]20D -3.4.degree. (2 min.) .fwdarw. -36.4.degree. (3 min.) .fwdarw. -52.degree. (10 min.) .fwdarw. -58.degree. (16 min.) .fwdarw. -58.degree. (35 min.) .fwdarw. -54.3.degree. (75 min.) .fwdarw. -9.1.degree. (9 hrs.) .fwdarw. 21.degree. (40 hrs.). To 3 g. prehydrogenated Pd(OH)2-BaSO4 (XXI) in 15 cc. H2O was added 2.5 g. VIII in 60 cc. 0.5N HCl, the mixt. hydrogenated 100 min. (H uptake, 2.2 mol. equivs.) and filtered, the soln. freed from Cl- by addn. of Ag2CO3 and filtered, the soln. freed from Ag+ with H2S, the filtrate evapd. in vacuo, and the residue crystd. from H2O-EtOH, to give 1 g. 2-amino-2-deoxy-D-galactonic acid (XXII), m. 198-203.degree. (decompn.), [.alpha.] 18D -4.95.degree. (c 0.6, H2O), -11.3.degree. (5 min.) .fwdarw. -31.degree. (24 hrs.; c 0.97, 2N HCl). To a suspension of 2 g. VIII in 25 cc. H2O was added 75 cc. 0.1N H2SO4 during 35 min.; after addn. of 65 cc., a clear soln. (pH 5) resulted, and addn. of the rest gave pH 3. During the addn., the content of NH4+ ions increased (Nessler reagent). The soln. was heated 90 min. (steam bath), evapd. in vacuo, the residue treated with 50 cc. MeOH-EtOH, the mixt. kept 12 hrs. at 0.degree., filtered, and the crystals washed with MeOH to give 0.36 g. (NH4)2SO4; the filtrate was hydrogenated as above and gave 0.9 g. XXII. Hydrolysis of VIII with dil. HCl gave only 50% NH4Cl. An aq. suspension of VIII was mixed with Ba(OH)2 soln. and gently warmed; VIII quickly dissolved and XIV soon crystd. The mixt. was filtered, the filtrate heated 20 min. (steam bath; HCN evolution), the Ba2+ ions pptd. with 2N H2SO4, the mixt. filtered, and the filtrate evapd. in vacuo to give a low yield of cryst. 2,3-dideoxy-2-phenylamino-D-threo-hexono-2-ene-1,4-lactone (XXIII). XI reduced cold NH4OH-AgNO3 soln. and Fehling soln. (60-70.degree.), but not Tillmans reagent. To a suspension of 10 g. XI in 50 cc.

H2O, 4 g. NaHCO3 was added and, dropwise, N iodine (uptake, 77 cc.). XI (20 q.) in 300 cc. satd. Ba(OH)2 was heated on the steam bath until the Prussian blue reaction was neg. (2 hrs.; NH3 evolution), the Ba2+ ions were pptd. with H2SO4, the mixt. filtered, 20 cc. concd. NH4OH added to the filtrate, the soln. evapd. in vacuo, the sirup boiled with 50 cc. MeOH, the crystals sepd. from mother liquor (XXIV), washed (EtOH), and recrystd. (90% aq. MeOH-Et2O) to give 5.9 g. NH4 2-deoxy-2-phenylamino-D-gulonate (XXV), m. 165.degree., [.alpha.] 20D 45.3.degree. (c 1.2, H2). XXV (3 g.) in 20 cc. N HCl was mixed with 3 g. XXI in 10 cc. H2O and hydrogenated 5 hrs. to give a sirup which was condensed with BzH-HCl to give 2-aminomono-O-benzylidene-2-deoxy-D-gulonolactone-HCl, m. 192.degree., [.alpha.]19D -58.degree. (c 0.88, 50% EtOH). XXIV was mixed with 100 cc. EtOAc to ppt. an oil which crystd. (2 days) and was recrystd. (95% EtOH, addn. of EtOAc) to give 2.3 g. NH4 2-deoxy-2-phenylamino-D-idonate (XXVI), m. 159-60.degree.,[.alpha.]18D -33.7.degree. (c 0.41, H20). XXVI (1.3 g.) was hydrogenated in acid soln. in the presence of 2 g. XXI to give 2-amino-2-deoxy-D-idonic acid, m. 230-6.degree. (decompn.) (from H2O-MeOH), (c 1.75, 25% HCl). A suspension of 10 g. XI in 30 cc. H2O was heated 3 hrs. on the steam bath, kept 3 hrs. at 0.degree., filtered, and the ppt. recrystd. from H2O (C) to give 0.7 g. XXIII, m. 142-4.degree.; the filtrate was treated with C, mixed with 1 cc. concd. NH4OH, and evapd. in vacuo, the residue treated with 50 cc. boiling EtOH, and the crystals fractionally recrystd. (90% MeOH; addn. of EtOAc) to give 2 g. XXV and 0.3 g. XXVI. g.), suspended in 20 cc. H2O, was dissolved by addn. of 40 cc. $exttt{N}$ HCl, the soln. (pH 4) added to 25 cc. 2N Na2CO3, the pptd. sirup crystd. by addn. of 5 cc. N NaOH, and the crystals washed with H2O to give 3.3 g. XIV, m. 142-3.degree.. XI (13 g.) was quickly dissolved in 100 cc. 2N H2SO4 and, after 30 min., the yellow ppt. was removed, washed with H2O and dried to give 4 g. 4,6-dihydroxy-2-phenylaminosorbic 1,4-lactone (XXVII), m. 104.degree. (MeOH-H2O), [.alpha.]D 0.degree., reduced Fehling soln., decompd. by boiling concd. HCl, no reaction with PhNHNH2, deep-red color with PhN2Cl (H2O-C5H5N.fwdarw.MeOH), no reaction with Ehrlich aldehyde reagent. XXVII (100 mg.) in 1 cc. C5H5N was treated with 0.5 cc. Ac2O to give 70 mg. 6-acetate of XXVII, needles (EtOH-H2O), m. 111.degree. A soln. of 2.3 g. XXVII in 70 cc. MeOH was mixed with 2.3 g. XXI in 25 cc. 1:5 H2O-MeOH and hydrogenated 2 hrs., to give 1.5 g. 4,6-dihydroxy-2-phenylaminohexanoic 1,4-lactone (XXVIII), needles, m. 134-5.degree. (MeOH). hydrogenation of XXVIII, the Ph group was hydrogenated; periodate oxidn. of the product (pH 11) gave Acetylation of XXVIII gave the 6-acetate, m. 108.degree. To a suspension of 0.5 g. XIV in 2 cc. H2O was added 4 (MeOH-H2O). cc. 0.5N H2SO4, the mixt. filtered, the soln. kept at 0.degree., and the crystals removed and washed (MeOH) to give 0.4 g. XIV.0.5H2SO4, decompg. at 140.degree. (browning, 115.degree.). Addn. of 2 cc. 2N HCl to a suspension of 1 g. XIV in 3 cc. H2O similarly gave 0.5 g. XIV.HCl, m. 83-7.degree.. Each salt regenerated XIV on treatment with 2N Na2CO3. To a suspension of XIV in 15 cc. H2O was added 15

cc. 2N HCl, and the soln. kept 2 days at 10.degree. to give 0.55 g. (crude) XXIII, m. 144.degree. (H2O), [.alpha.]20D -181.degree. (c 0.68, MeOH), reduced Fehling soln. A suspension of 20 g. XIV in 200 cc. H2O was heated 90 min. (steam bath), decanted hot from about 3 q. oil, and the soln. cooled to give 5 g. XXIII, m. 144.degree.. Acetylation of XXIII with Ac20-C5H5N gave XXIII diacetate (XXIX), needles, m. 87.degree. (MeOH + H2O), [.alpha.]19D -124.degree. (c 0.95, MeOH), reducing to Fehling soln. XXV (3 g.) and 1 cc. concd. HCl was evapd. to dryness in vacuo, the residue mixed with 20 cc. Ac20 and 3 g. anhyd. NaOAc, the mixt. boiled 1 min., cooled, treated with 10 cc. H2O and then with satd. Na2CO3 until neutral, and the pptd. sirup rubbed with H2O to give 0.8 g. cryst. XXIX. VIII (5 q.) was hydrolyzed with dil. H2SO4, and the (NH4)2SO4 removed with EtOH to give 2.3 g. sirupy N-Ph deriv. (XXX) of XXII; XXX was treated (as for XXV .fwdarw. XXIX) to give 0.3 g. XXIX. A suspension of 0.64 g. XXIII in 40 cc. H2O was mixed with 2 g. NaIO4 in 10 cc. H2O, the mixt. kept 1 hr. in the dark at 20.degree., filtered, acidified (dil. HCl), the excess periodate destroyed with NaAsO2, the soln. buffered with NaOAc, filtered, the soln. mixed with a soln. of dimedon in EtOH, and the mixt. kept 3 hrs. to give 0.44 g. dimedon-HCHO adduct. XXIII (6 g.) in 200 cc. anhyd. MeOH at -80.degree. was treated 70 min. with 5-6% O3-O2 (12 l./hr.), the product hydrogenated in the presence of 3 g. prehydrogenated PdO in 20 cc. anhyd. MeOH (H uptake, 640 cc. at 20.degree./752 mm.), the mixt. filtered, the filtrate evapd. in vacuo, the residue dissolved in 10 cc. H2O, and extd. with three 15-cc. portions 1:1 EtOAcEt20 (XXXI) to give an aq. (A) and an org. (B) phase. A was evapd. in vacuo to give 2.5 g. sirupy, impure D-threose, descending papergram Rf 0.65 (PhNH2 H phthalate and periodate-benzidine sprays). B was dried and evapd., the residue mixed with aq. Ca(OAc)2 soln., the mixt. boiled, cooled, and filtered, the ppt. (Ca salt) treated with N H2SO4, filtered, the soln. extd. with XXXI, the ext. dried, evapd., and the residue crystd. (C6H6) to give oxanilic acid. A soln. of 0.6 g. XXIII in 25 cc. MeOH was mixed with 1 g. XXI in 20 cc. MeOH and hydrogenated 30 min. the mixt. filtered, the soln. evapd., the residue dissolved in EtOAc, the soln. treated with dry HCl, and the ppt. crystd. from 3 cc. EtOH to give 0.2 g. 2,3-dideoxy-2phenylamino-D-lyxo (or D-xylo)-hexonic 1,4-lactone hydrochloride, needles, m. 158-62.degree., [.alpha.]19D -34.degree. (c 1.1, H2O), nonreducing to Fehling soln. A mixt. of 3.4 g. XXIII with 30 cc. 0.5N HCl and 4 q. XXI in 10 cc. H2O was hydrogenated 21 hrs. (H uptake, 2.76 molar equivs.), the mixt. filtered, the soln. evapd. and the residue crystd. (EtOH; then 90% aq. MeOH-EtOAc) to give 1.2 g. 2-amino-2,3-dideoxy-D-lyxo (or D-xylo)-hexonic 1,4-lactone hydrochloride (XXXII), m. 202-3.degree., [.alpha.]22D -31.9.degree. (c 0.76, H2O). XXXII was reduced with NaHg-H2SO4 to V (D-lyxo or D-xylo) hydrochloride, m. 146.degree., [.alpha.]20D 60.2.degree. (3 min.) .fwdarw. 23.9.degree. (4 hrs.; c 0.83, H2O), Rglucosamine 1.05. Action of warm dil. acids on XIV or XXIII gave the 1,4-lactone (XXXIII) of VI (D-threo) (XXXIV). An aq. soln. of XXXIV was treated 12 hrs. with excess o-C6H4(NH2)2 to give the

quinoxaline deriv. of XXXIV m. 160-61.degree. (EtOH), [.alpha.]20D 39.4.degree. (c 1.01, MeOH). A soln. of 0.7 g. XXXIII in 3 cc. H2O was treated with 10 drops 30% H2O2 and the mixt. warmed to 50.degree. (CO2 evolution) and, finally, boiled 1 min. A papergram showed no XXXIII, but 2 new, faster-moving spots. The soln. was evapd. in vacuo, the residue heated 1 min. at 125.degree. with 1 cc. PhNHNH2, and the product digested with EtOAc to give 0.15 g. phenylhydrazide (XXXV) of 2-deoxy-D-threo-pentonic acid (XXXVI), m.p. and mixed m.p. 138.degree.. Sirupy XXXIII was mixed with an equal wt. of PhNHNH2, the mixt. heated 2 min. on the steam bath, treated with H2O, and the mixt. filtered to give the phenylhydrazone (XXXVII) of XXXIII, yellow leaflets, m. 213-14.degree. (EtOH or EtOAc), [.alpha.]18D -270.degree. (c 0.45, C5H5N). The same compd. resulted on short heating of XIV or XXIII with 50% HOAc and treatment with PhNHNH2, or by treating with PhNHNH2 in 50% HOAc at 70-80.degree.. Attempts to split the hydrazone with BzH or AcCO2H Treatment of XXXVII with Ac2O-C5H5N 2 min. at 65.degree. gave a quant. yield of diacetate of XXXVII, prisms, m. 188.degree. (10:1 EtOHMe2CO, plus petr. ether), [.alpha.]20D -137.degree. (c 0.76, C5H5N). A soln. of XXXVII and excess p-O2NC6H4CHO in PrOH was boiled 4 hrs. to give the p-nitrobenzylidene deriv., yellow needles, m. 245.degree. (C5H5N-H2O), [.alpha.]19D -138.degree. (c 0.38, Hydrogenation of XXXVII in aq. ethanolic HCl in the presence of XXI, with uptake of 2 molar proportions H, gave 20-30% XXXII, m. 203.degree., [.alpha.]20D -31.degree. (c 0.96, H2O). XIV was treated with PhNHNH2 in 50% HOAc, the ppt. was removed, dried, extd. with Et20 and with Et0Ac, the combined exts. evapd., the residue dissolved in EtOH and treated with H2O to give the phenylhydrazide phenylhydra-zone (XXXVIII) of XXXIII, yellow needles, m. 204-5.degree., [.alpha.]18D 13.9.degree. (c 0.57, C5H5N). Hydrogenation of XXXVIII as for XXXVII gave XXXII. A suspension of 6 g. XIV in 200 cc. anhyd. MeOH was ozonized 1 hr. (O speed, 14 l./hr.; 5-6% O3), the insol. matter (about 1 g.) removed, the soln. hydrogenated (Pd; H uptake, 280 cc.), the mixt. filtered, and the filtrate evapd. to give crude D-threose, identified by Rf on a papergram. A mixt. of 10 g. XIV and 120 cc. 0.3N Ba(OH)2 was heated 5 hrs. on a steam bath, the insol. residue removed, the soln. passed through Amberlite IR-120, the column eluted with 200 cc. H2O, the combined eluates evapd. in vacuo, the sirup (4.3 g.) dissolved in 25 cc. H2O, and 0.3 cc. PhNHNH2 added to give 0.6 g. XXXVII; the mother liquor was re-treated with Amberlite IR-120, the combined eluates evapd., the residue (2.9 g.) dissolved in 1:2 EtOH-Me2CO, the mixt. filtered, and the soln. mixed with 10 vols. petr. ether, to give 1.5 g. anilide (XXXIX) of XXXVI, m. 118.degree. (EtOH-petr. ether), [.alpha.] 19D 31.3.degree. (c 0.96, H2O). XXXIX was treated with boiling 2N NaOH, the mixt. cooled and extd. with Et20, the aq. soln. evapd., and the residue treated with MeOH to give the Na salt, needles, m. 183.degree., [.alpha.]18D 12.1.degree. (c 1.24, H2O). A mixt. of 1 g. XXXIX and 2 cc. 2N KOH was heated 1 hr. on a steam bath, the K+ ions removed with Amberlite IR-120, the eluate evapd., the residue heated 5 min. at 130.degree., cooled to 80.degree., 1 cc. PhNHNH2 added, the mixt. kept 5 min. at

130.degree., cooled, washed with abs. Et20, and the residue crystd. by brief boiling with EtOAc to give 0.23 g. XXXV, m. 139.degree. (EtOH + Et2O), [.alpha.] 19D 8.8.degree. (c 1.6, MeOH). similar to those used for XIV, XVII gave similar products; XVII.HCl, colorless needles, m. 76-7.degree.. Treatment of XVII with dil. HCl 2 days at room temp. gave X (D-erythro) (XL), prisms, m. 134.degree. (H2O), [.alpha.]20D 115.degree. (c 0.92, MeOH). A mixt. of 1.8 g. XL, 30 cc. H2O, 4 cc. 2N HCl, and 2 g. XXI was hydrogenated 5 hrs. (uptake, 3.2 molar equivs. H), the mixt. filtered, the soln. evapd., and the sirup crystd. (EtOH) to give 0.7 g. D-arabino or D-ribo isomer (XLI) of XXXII, prisms, m. 194.degree. (EtOH-EtOAc), [.alpha.]20D -7.8.degree. (c 2.04, H2O). Hydrolysis of XL with Amberlite IR-120, as for XXIII, gave 70% sirupy VI (D-erythro) (XLII), [.alpha.]22D -34.5.degree. (c 41.3, H2O), Rf 0.21; quinoxaline deriv. m. 174.degree. (EtOH-EtOAc), [.alpha.]19D -58.4.degree. (c 0.48, MeOH). XLII was converted to the D-erythro isomer (XLIII) of XXXV, m. 145.degree.. A mixt. of 1 g. XVII, 50 cc. 50% HOAc, and 1 cc. PhNHNH2 was kept 20 min. at 60-70.degree., the soln. poured into 50 cc. H2O, and the ppt. washed with H2O and crystd. from H2O to give 0.4 g. D-erythro isomer (XLIV) of XXXVII, yellow needles, m. 229.degree. (decompn.), [.alpha.]19D 168.degree. (c 2.47, C5H5N). A mixt. of 1 g. XLII, 100 cc. EtOH, 10 cc. 2N HCl, and 1 g. XXI in 15 cc. H2O was hydrogenated 14 hrs. (H uptake, 2.3 molar equivs.), 15 cc. H2O added, the suspension centrifuged, the soln. evapd. in vacuo, the residue twice extd. with abs. EtOH and twice crystd. from MeOH to give 80 mg. XLI, needles, m. 195.degree., [.alpha.]22D -7.5.degree. (c 1.6, H20). XVII was treated 1-2 hrs. with Ba(OH)2 soln. at 45-55.degree. as for XIV, the warm mixt. filtered, the soln. cooled, and the crystals recrystd. (H2O or MeOH); the mother liquor was reheated 10 min., freed from Ba2+ ions with 2N H2SO4, the hot suspension filtered, and the filtrate cooled to give a second crop (total, 55%) of D-erythro isomer (XLIV) of XXXIX, leaflets, m. 187.degree. (MeOH), [.alpha.]20D -31.8.degree. (c 0.85, MeOH). From the mother liquor, some XLII could be pptd. as the Ca salt.

XLIV was boiled 90 min. with 2N KOH, the soln. cooled, passed through a column of Amberlite IR-120, the eluate evapd., the oil kept 10 min. at 130-40.degree., mixed with an equal wt. of PhNHNH2, the mixt. kept 5 min. at 130-40.degree., cooled, washed with Et2O, and rubbed with EtOAc to give XLIII, needles (MeOH-Et2O), m. 149.degree., [.alpha.]19D -11.degree. (c 1.28, MeOH). The infrared spectra of XVIII, VIII, XI, XIV, XXIII, XXVII, and XXXVIII, and the ultraviolet spectra of XXVII, 2-acetamido-4,6-dihydroxysorbic lactone, and 3-acetamido-6 (acetoxymethyl)-2H-pyran-2-one were recorded.

RN 23276-49-5 HCAPLUS

CN 2(1H)-Quinoxalinone, 3-(2,3,4-trihydroxybutyl)-, [S-(R*,S*)]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

RN 122240-52-2 HCAPLUS CN Threitol, 1-deoxy-1-(3-hydroxy-2-quinazolinyl)-, D- (6CI) (CA INDEX NAME)

Absolute stereochemistry.

10C (Organic Chemistry: Carbohydrates, Amino Acids, and Proteins) CC488-32-4, Idonic acid, 2-amino-2-deoxy-, D- 17510-99-5, IT D-erythro-Hexulosonic acid, 3-deoxy- 23276-49-5, Erythritol, 1-deoxy-1-(3-hydroxy-2-quinoxalinyl)-, D-100063-47-6, Sorbic acid, 2-anilino-4,6-dihydroxy-, .gamma.-lactone 107036-82-8, D-ribo-Hexonic acid, 2-amino-2,3-dideoxy-, .gamma.-lactone, hydrochloride 107036-85-1, D-lyxo-Hexonic acid, 2-amino-2,3-dideoxy-, .gamma.-lactone, hydrochloride 108595-07-9, Mannononitrile, 2-anilino-2-deoxy-, D- 108595-27-3, D-xylo-Hexofuranos-1-enylamine, 2-anilino-2-deoxy-108629-97-6, D-erythro-Hexulosonic acid, 3-deoxy-, .gamma.-lactone, 108846-30-6, D-xylo-Hexonic acid, phenylhydrazone 2-anilino-2,3-dideoxy-, .gamma.-lactone, hydrochloride 109047-67-8, D-erythro-Hexon-2-enic acid, 2-anilino-2,3-dideoxy-, .gamma.-lactone 109062-47-7, D-lyxo-Hexonic acid, 2-anilino-2,3-dideoxy-, .gamma.-lactone, hydrochloride 109188-62-7, Gulononitrile, 2-anilino-2-deoxy-, D-109188-63-8, Idononitrile, 2-anilino-2-deoxy-, D- 109188-64-9, Talononitrile, 2-anilino-2-deoxy-, D- 109256-98-6, Galactononitrile, 109256-99-7, Altrononitrile, 2-anilino-2-deoxy-, D-109367-09-1, Norleucine, 2-anilino-2-deoxy-, D-4,6-dihydroxy-N-phenyl-, .gamma.-lactone 110533-61-4, D-erythro-Pentonic acid, 2-deoxy-, phenylhydrazide 112072-00-1, D-threo-Hexulosonic acid, 3-deoxy-5,6-0-p-nitrobenzylidene-,

114843-22-0, D-lyxo-Hexose, 2-amino-2,3-dideoxy-, phenylhydrazone 114843-23-1, D-xylo-Hexose, 2-amino-2,3-dideoxy-, hydrochloride 118101-15-8, D-erythro-Pentonanilide, 2-deoxyhvdrochloride 118101-16-9, D-threo-Pentonanilide, 2-deoxy-119149-47-2, D-arabino-Hexonic acid, 2-amino-2,3-dideoxy-, .gamma.-lactone, 119149-48-3, D-xylo-Hexonic acid, hydrochloride 2-amino-2,3-dideoxy-, .gamma.-lactone, hydrochloride 122147-13-1, D-threo-Hexon-2-enimidic acid, 2-anilino-2,3-dideoxy-, 122147-14-2, D-erythro-Hexon-2-enimidic acid, .gamma.-lactone 2-anilino-2,3-dideoxy-, .gamma.-lactone 122240-52-2, Threitol, 1-deoxy-1-(3-hydroxy-2-quinazolinyl)-(?), D-(prepn. of)

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ACCESSION NUMBER: 1961:124877 HCAPLUS

55:124877 DOCUMENT NUMBER:

ORIGINAL REFERENCE NO.: 55:23539q-i,23540a-f

1,4-Oxazines. III. The condensation of TITLE:

bifunctional .alpha.-oxo esters with

o-amlnophenols and o-phenylenediamines to

symmetrical cyanines

Biekert, Ernst; Enslein, Lore AUTHOR(S):

Max-Planck-Inst. Biochem., Munich, Germany CORPORATE SOURCE:

Chemische Berichte (1961), 94, 1851-60 SOURCE:

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE:

Journal

Unavailable LANGUAGE: The condensation of o-aminophenols with cf. CA 55, 19928h. AΒ .alpha.-oxo esters to 1,4-benzoxazin-2-ones was extended to The bis(1,4-benzoxazin-2-on-3bifunctional .alpha.-oxo esters. yl)acetones obtained from CO(CH2COCO2Et)2 (I) were high-melting, deeply colored compds. with a sym. cyanine structure; with o-phenylenediamines similar cyanine-like compds. of deep color were formed. 4,6,1,3-(O2N) 2C6H2(OH) 2 in EtOH hydrogenated over Raney Ni and added under N to an equiv. amt. of EtO2CCOCH2CO2Et gave about 50% benzobis(1,4-oxazin-2-one), m. 270-5.degree. (PhCl), with slow darkening. I (600 mg.) and 200 mg. o-H2NC6H4OH (II) in cold MeOH refluxed 0.5 hr. with 1 drop 2N HCl and concd. gave about 75% Et 5-(1,4-benzoxazin-2-on-3-yl)-2,4-dioxovalerate (III), orange needles, m. 200.degree. (MeOH). A similar run during 1 day at room temp. gave about 50% III; in refluxing BuOH the reaction was completed after several min. 2,3,5-H2NMe2C6H2OH (IV) (66 mg.) and 132 mg. I in 5 cc. MeOH heated briefly to 50.degree. yielded about 60% Et 5-(5,7-dimethyl-1,4-benzoxazin-2-on-3-yl)-2,4-dioxovalerate (V), orange needles, m. 248.degree. (BuOH). I and 2,4,6-H2NMe2C6H2OH (VI) gave similarly 75-80% 6,8-di-Me isomer of V, orange needles, decompg. 236.degree. (Me2CO). 2,3-H2NC10H6OH (VII) in about 10 cc. boiling MeOH added to 224 mg. I in MeOH and kept 6 days at room temp. yielded 67% 5-(naphtho[2',3':5,6]-1,4-oxazin-2-on-3-yl) analog of III, orange-red needles, decomp. 243.degree. (CHCl3). I (2.58 g.) and 2.18 g. II heated 10 min. at 165.degree. and boiled several times with MeOH left about 60% dark red

1,3-bis(1,4-benzoxazin-2-on-3-yl)acetone (VIII), decompg. about 305.degree. (HCONMe2); a similar run at room temp. during 6 weeks gave about 50% VIII; in refluxing BuOH 60% VIII was obtained. (80.5 g.) and 91 g. I in 1.5 l. C6H6 refluxed 5 hrs. gave 117 g. VIII, decompg. 300.degree.. I (133 mg.) and 152 mg. IV in MeOH evapd. and heated to 150.degree. gave 67% bis(5,7-dimethyl-1,4benzoxazin-2-on-3-yl)-acetone (IX), violet needles, decompg. 330.degree. (HCONMe2). VI gave similarly the 6,8-di-Me isomer of IX, decompg. 319.degree. (HCONMe2). III (100 mg.) and 50 mg. IV in a little hot HCONMe2, slowly distd. gave 75% 1-(1,4-benzoxazin-2-on-3-y1)-3-(5,7-dimethyl-1,4-benzoxazin-2-on-3-y1)acetone (X), red-violet needles, decompg. 293.degree. (HCONMe2). heated at 160-80.degree. without solvent gave the bis(naphtho-[2',3';5,6]-1,4-oxazin-2-on-3-yl)acetone, decompg. 318.degree. (dioxane). VIII (2 g.) added during 1 hr. with stirring at room temp. to about 10 cc. 20% oleum, stirred to soln., dild. with stirring and cooling slowly with 200 cc. dry Et-OAc, kept several hrs. at 0.degree., and centrifuged gave the very hygroscopic di-SO3H deriv. of VIII, did not melt up to 360.degree.. o-C6H6(NH2)2 (1.1 q.) in the min. amt. MeOH added dropwise to 2.8 g. I in 25 cc. hot 90% MeOH, refluxed 2.5 hrs., and filtered yielded 1.05 g. Et 5 (4-aza-3-coumarinyl)-2,4-dioxovalerate (XI), dark red, decompg. 248.degree. (HCONMe2); the mother liquor gave 1 g. brown red needles, m. 147.degree., which were not further investigated. XI (433 mg.) and 155 mg. II heated 20 min. at 170.degree., cooled, and boiled with MeOH gave 445 mg. 1-(4-aza-3-coumarinyl)-3-(quinoxal-3-on-2-yl)acetone, red needles, decompg. 305.degree. (HCONMe2-EtOH). o-H2NC6H4NHPh (XII) (560 mg.) in BuOH added to 800 mg. I in BuOH and refluxed 0.5 hr. gave 380 mg. 4-Ph deriv. of XI, decompg. 225.degree. (BuOH). XII (8 g.) and 5.2 g. I heated 1 hr. under N at 110-20.degree., cooled, and dild. with MeOH, and the cryst. ppt. boiled with MeOH yielded 3.5 g. 1,3-bis(4-phenylquinoxal-3-on-2yl)acetone, decompg. 281.degree. (BuOH). The ultraviolet absorption spectrum of VIII and the infrared absorption spectra of III and VIII were recorded.

1T 101884-52-0, 2H-1,4-Benzoxazin-2-one, 3-[3-(3,4-dihydro-3-oxo-2-quinoxalinyl)acetonyl]- 102311-83-1,
2-Quinoxalinevaleric acid, 3,4-dihydro-.alpha.,.gamma.,3-trioxo-4-phenyl-, ethyl ester 106596-07-0, 2-Quinoxalinevaleric acid, 3,4-dihydro-.alpha.,.gamma.,3-trioxo-, ethyl ester 115759-36-9, 2(1H)-Quinoxalinone, 3,3'-(2-oxotrimethylene)bis[1-phenyl-(prepn. of)

101884-52-0 HCAPLUS

RN

CN

2H-1,4-Benzoxazin-2-one, 3-[3-(3,4-dihydro-3-oxo-2-quinoxalinyl)acetonyl]- (6CI) (CA INDEX NAME)

RN 102311-83-1 HCAPLUS

CN 2-Quinoxalinevaleric acid, 3,4-dihydro-.alpha.,.gamma.,3-trioxo-4-phenyl-, ethyl ester (6CI) (CA INDEX NAME)

RN 106596-07-0 HCAPLUS

CN 2-Quinoxalinevaleric acid, 3,4-dihydro-.alpha.,.gamma.,3-trioxo-, ethyl ester (6CI) (CA INDEX NAME)

$$\begin{array}{c|c} & \circ & \circ & \circ \\ \parallel & \parallel & \parallel \\ N & CH_2-C-CH_2-C-C-OEt \\ N & O & \\ N & H & O & \\ \end{array}$$

RN 115759-36-9 HCAPLUS

CN 2(1H)-Quinoxalinone, 3,3'-(2-oxotrimethylene)bis[1-phenyl-(6CI) (CA INDEX NAME)

$$\begin{array}{c|c} N & O \\ | \\ CH_2 - C - CH_2 \\ \hline N & O \\ \hline Ph & Ph \end{array}$$

CC 10G (Organic Chemistry: Heterocyclic Compounds)

101098-00-4, 2H-1,4-Benzoxazine-3-valeric acid, .alpha.,.gamma.,2-ITtrioxo-, ethyl ester 101884-52-0, 2H-1,4-Benzoxazin-2-one, 3-[3-(3,4-dihydro-3-oxo-2-quinoxalinyl)acetonyl]-102241-60-1, 2H-1,4-Benzoxazin-2-one, 5,7-dimethyl-3,3'-(2-oxotrimethylene)bis-102311-83-1, 2-Quinoxalinevaleric acid, 3,4-dihydro-.alpha.,.gamma.,3-trioxo-4-phenyl-, ethyl ester 106596-07-0 , 2-Quinoxalinevaleric acid, 3,4-dihydro-.alpha.,.gamma.,3-trioxo-, ethyl ester 111475-91-3, 2H,8H-Benzo[1,2-b:5,4-b']bis[1,4]oxazine-3,7-diacetic acid, 3,4,6,7-tetrahydro-2,8-dioxo-, diethyl ester 111664-72-3, 2H-Naphth[2,3-b]-1,4-oxazine-3-valeric acid, .alpha.,.gamma.,2-trioxo-, ethyl ester 112551-64-1, 2H-1,4-Benzoxazin-2-one, 3,3'-(2-oxotrimethylene)bis[6,8-dimethyl-112552-00-8, 2H-1,4-Benzoxazin-2-one, 3,3'-(2oxotrimethylene)bis[5,7-dimethyl- 115759-36-9, 2(1H)-Quinoxalinone, 3,3'-(2-oxotrimethylene)bis[1-phenyl-124121-76-2, 2H-Naphth[2,3-b]-1,4-oxazin-2-one, 3,3'-(2oxotrimethylene)bis-(prepn. of)